

# **COST REDUCTION IN BIO-DIESEL PRODUCTION**

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## SUMMARY

As part of a programme to assess bio-diesel production from low-cost materials, the availability of waste oils and fats in Ireland and the EU was assessed, and the behaviour of their esters in vehicles was measured. The utilisation of beef tallow from BSE risk organisms was given special attention.

Esterification of high-FFA tallow required the use of excess methanol and base catalyst. Acidification, followed by glycerol separation and secondary acid-catalysed esterification, brought ester yields up to acceptable levels. The high melting point of the ester restricted its use in vehicles to low proportions in mixes with mineral diesel.

Fuels made up from low-cost esters behaved well in vehicle trials, even where there was a high level of dilution of the engine oil. The only problems arose from inadequate low-temperature properties and from suspended solids in the tallow ester.

Within the EU, there are sufficient waste oils and fats available to greatly increase bio-diesel production. In Ireland, there is sufficient for a small production unit.

## INTRODUCTION

Rape methyl ester is now well proven as a fuel for diesel engines, mixed with mineral diesel or as a complete replacement. Engine power and fuel consumption are largely unaffected, and the fuel has some desirable environmental features: lower smoke, particulate levels and sulphur, and very low toxicity to water life. However, the high cost of rape-seed oil is a major problem. Lower-cost feedstocks are essential to the further growth of the industry.

In a parallel project (ARMIS No. 4355), bio-diesel from *Camelina sativa* oil was found to have acceptable properties, with the exception of a high iodine value. Esters of waste cooking oil had more variable properties than those from virgin oil, but still came close to conformity

with a draft bio-diesel standard<sup>1</sup>. Preliminary trials with tallow indicated low yields and poor low-temperature properties.

The present project had three objectives:

- To further explore the possibility of producing bio-diesel from tallow, in particular that from organisms considered to carry a risk of BSE transmission. At present this material has no other market and a high disposal cost.
- To examine the behaviour in vehicles of bio-diesel produced from alternative low-cost feedstocks.
- To estimate the amount of waste vegetable oil and tallow that might be available for bio-diesel production in Ireland and the European Union.

## **METHODS**

Tallow from BSE risk organisms (brain and spinal tissue) was esterified in the laboratory. Free fatty acid content was 15-20%, so esterification using the standard base-catalysed process was not possible. The process was adapted to the esterification of this material by using large excesses of base (+75%) and methanol (+50%), and using water to help with the separation of glycerol. Further process steps to improve yield were explored.

Three light vehicles were operated on five bio-diesel blends based on three feed-stocks: camelina oil, waste cooking oil and tallow. Vehicle monitoring included engine lubricating oil condition (methyl ester content, viscosity and wear metals), fuel consumption and practical observations of vehicle behaviour.

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<sup>1</sup> Commission of European Communities, 1993. Proposal for a Council Directive concerning the specifications for vegetable oil methyl esters as a motor fuel. May, 1993.

The quantities, availability and suitability as bio-diesel feedstocks of waste oils and fats in Ireland and the EU were assessed. The EU information was based on detailed reports from four countries: Austria, Belgium, Germany and Ireland.

## RESULTS AND DISCUSSION

### Tallow esterification

With the modified esterification process, it was possible to obtain a methyl ester which met all bio-diesel specifications with two exceptions:

1. Glyceride level slightly over specification (Table 1)
2. CFPP (cold filter plug point) about 17°C.

**Table 1:** Characteristics of tallow methyl esters

Sample	1	2	3	4	5	Spec.
Yield (% w/w)	55.7	55.6	55.8	55.8	55.3	
Ester properties:						
Acid value (mg KOH/g)	0.25	0.25	0.20	0.17	0.22	<0.5 <sup>1</sup>
Iodine value (mg I <sub>2</sub> /g)	51.1	52.7	-	-	-	<115 <sup>1</sup>
Viscosity 20°C (cSt)	8.17	8.23	8.18	8.15	8.16	6.0-9.0 <sup>2</sup>
Free glycerine (% w/w)	0.003	0.002	0.003	0.002	0.001	<0.03 <sup>1</sup>
Triglycerides (% w/w)	nd	nd	nd	Nd	Nd	
Diglycerides (% w/w)	2.0	1.47	-	1.91	1.55	
Monoglycerides (% w/w)	0.53	0.44	-	0.50	0.45	<0.8 <sup>1</sup>
Bound glycerine (% w/w)	0.45	0.35	-	0.43	0.36	<0.2 <sup>1</sup>
Total glycerine (% w/w)	0.45	0.35	-	0.43	0.36	<0.25 <sup>1</sup>
Methanol (% w/w)	nd	nd	nd	Nd	Nd	<0.30 <sup>1</sup>

1. Commission of European Communities, 1993. Proposal for a Council Directive concerning the specifications for vegetable oil methyl esters as a motor fuel. May, 1993.
2. Ö Norm C1190, 1995. Vornorm-Kraftstoffe – Dieselmotoren, rapsolmethylester, anforderungen. Vienna.

The latter problem arises from the high proportion of saturated fatty acids in the tallow. Previous work suggested that the only way of using this material as a vehicle fuel is in blends of 10% or less with mineral diesel. In this application, the measured glyceride levels would not present a problem.

Methyl ester yields obtained with this process were about 56% (w/w). The low yield was caused by the high concentration of potassium salts of free fatty acids (soaps) which dissolved over 25% of the methyl ester. It appeared that yields could be improved considerably by acid-catalysed esterification of the fatty acids recovered from the glycerol phase. The second esterification could be included as an extra processing step, with no need to isolate the glycerol beforehand.

After base-catalysed esterification, the glycerol-methyl ester mixture was acidified to remove the fatty acids and methyl ester from the glycerol. Glycerol was removed at this stage, and the organic phase, which contained the methyl ester and free fatty acids produced in the first esterification, was esterified with methanol and an acid catalyst. The catalysts used were boron trifluoride, hydrochloric acid and sulphuric acid. All three reduced free fatty acid levels below specification, and the final yields based on tallow were over 98% (w/w). The results obtained showed that by adding two simple steps to the standard esterification process, high yields of methyl ester could be obtained from low-grade tallow.

## Vehicle trials with bio-diesel from low-cost feedstocks

The blends chosen for the vehicle performance tests are listed in Table 2. Fuels 1 to 4 were chosen to give a CFPP of about  $-3^{\circ}\text{C}$ . A CFPP level above the minimum expected ambient temperature was chosen so that the relationship between fuel CFPP and vehicle starting problems could be explored. Fuel 1, with an iodine value of 155, was chosen to examine the effect of high iodine value on lubricating oil performance. Fuel 2 was chosen to maximise the use of camelina in a blend with 115 iodine value. Fuels No. 3 and 4 were selected to maximise tallow use, one with vegetable oils and the other with mineral diesel. Fuel No. 5 allowed for substantial use of tallow and waste cooking oil esters in a blend suitable

for year-round use in Ireland. All blends were used for at least one oil change interval.

**Table 2:** Fuel blends and use periods in vehicle performance trials

Fuel no.	Composition	CFPP (°C)	Iodine value	Period of use (1996-97)
1	100% CME	-3	155	Sept. 27 - Feb. 6
2	5% TME 30% CME, 65% WME	-3	115	Feb. 7 - June 23
3	5% TME 95% WME	-3	97.5	Sept. 27 – Jan. 9
4	30% TME 70% diesel	-3	17	Oct. 18 - July 29
5	10% TME 40% WME 50% diesel	-8	48	April 7 - July 18

The vehicles used in the trials were a Peugeot 306 Xad with a 1905cc indirect injection engine, an Isuzu Trooper UBS55 with a 2800cc direct injection turbo-charged engine, and a Toyota Dyna 100 with a 2400cc indirect injection engine. The distances travelled and fuel consumed by each vehicle in unreplicated comparisons during routine work were as in Table 3.

In all cases, fuel economy was slightly lower with the bio-fuels than with mineral diesel, the difference being from 1.5 to 6%. Differences between the bio-diesel blends were small.

Four of the fuel blends were also compared in replicated (3 replications) trials over two road circuits of 58 and 101 km respectively in the Peugeot 306. The results of these trials are given in Table 4.

**Table 3:** Fuel economy during routine work with bio-diesel blends in three vehicles

Vehicle	Fuel no.	Distance travelled (km)	Fuel economy (km/litre)
Peugeot	3	5753	16.34
	5	7877	15.92
	mineral diesel	16268	16.92
Isuzu	1	7509	10.92
	2	7037	11.01
	mineral diesel	8915	11.18
Toyota	4	8101	9.66
	mineral diesel	3394	10.28

**Table 4:** Fuel economy in replicated trials of four bio-diesel blends and mineral diesel

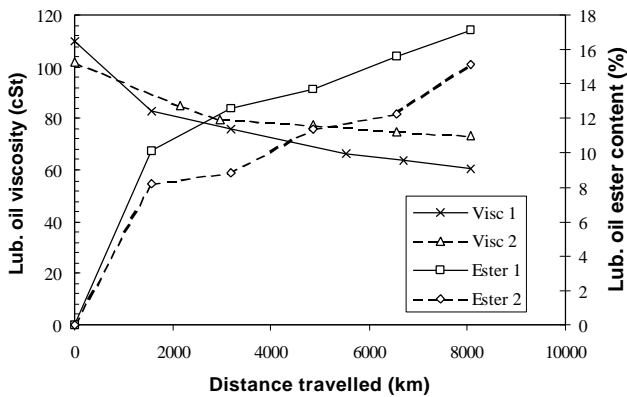
Circuit	Speed (km/hr)	Fuel no.	Fuel economy (km/litre)	F-value (sign.)
1	87.5	1	17.61	1.32 (n.s.)
	89.7	2	17.16	
	88.6	4	17.71	
	88.5	5	17.59	
	85.6	mineral	17.88	
2	86.0	1	18.06	0.64 (n.s.)
	85.9	2	18.10	
	83.3	4	18.30	
	83.7	5	18.06	
	83.2	mineral	18.93	

Results for the two circuits were similar, and both were similar to the unreplicated trial; fuel economies with the bio-diesel blends were from 1.5 to 4% lower than with mineral diesel, and differences between blends were small. None of the differences in the trial reached statistical significance.

The Isuzu Trooper was the only vehicle in which there was a significant accumulation of ester in the lubricating oil, reaching about 16% with Fuels 1 and 2 at the end of the trial (Fig. 1). This allowed a comparison to be made of the effects of high and low iodine value esters on lubricating oil performance in conditions of high dilution.



Lubricating oil viscosity changes followed similar patterns with both fuels. The ester dilution was slightly greater for Fuel 1, as a result of which the fall in viscosity was greater. However, there was no evidence of any adverse effect of the high iodine number of Fuel 1 on the properties of the lubricating oil.



**Fig. 1:** Variation in lubricating oil ester content and viscosity at 40°C with two bio-diesel fuels over one oil-change interval

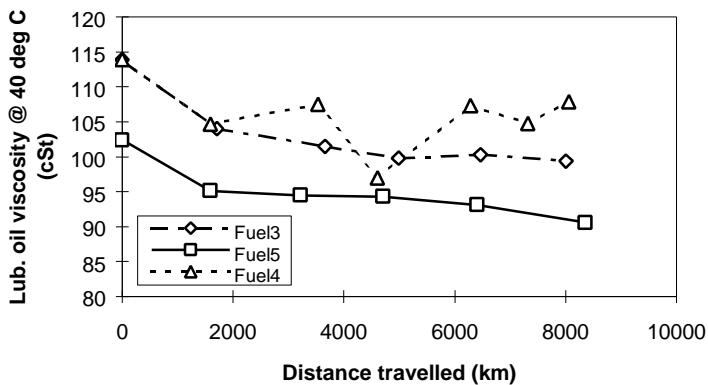
In the other two vehicles, there was no detectable dilution of the lubricating oils with methyl ester, and the viscosity changes were typical of the results one might expect with mineral diesel (Fig. 2).

Wear metal levels in the lubricating oils were also within normal limits, with the exception of a very high copper level in the final sample removed from the Peugeot with Fuel 5. This is not thought to have been related to the fuel use.

The only operational problems of significance to affect the vehicles in the course of the trials were as follows:

- (i) *Inadequate low-temperature properties:* Fuels 1 and 3 were in use during the coldest period of the winter, when temperatures were occasionally below the CFPP of the fuels. This led to starting difficulties and fuel filter blockages.

- (ii) *Inadequate filtration after esterification:* All the fuels containing tallow ester gave some problems with fuel filter blockages. This was apparently due to the presence of suspended solids which were not fully removed by filtering of the tallow ester at high temperatures immediately after drying. When each fuel blend was filtered at ambient temperature through a 10-micron filter, the fuel filter blockage problems did not recur.



**Fig.2:** Viscosity variations with three fuels in which no ester dilution was detected

### Potential of waste oils and fats as bio-diesel feedstocks in Ireland and the EU

The reported rate of consumption of vegetable oils was 20-30 kg/head in Austria, Germany and Ireland, but 40 kg/head in Belgium. The average for the EU was about 35 kg/head. There was a wide range of variation in animal fat consumption, from 21 kg/head in Belgium to about 5 kg/head in Germany and Ireland.

In Austria, the amount of collectible waste vegetable oil has been estimated at 37,000 t, or 4.6 kg/head. The amounts actually collected in each country are estimated at 1.1 to 1.6 kg/head. If the quantities currently recovered throughout the EU are close to this figure, the

total amount collected in the EU would be about 560,000 tonnes. Extrapolating the potential percentage recovery estimated by the four countries in this survey (about twice the present recovery) to the total community, the total volume of recoverable oil would be over 1 million tonnes. Given that this is considerably more than the consumption of the existing bio-diesel industry, its potential to support further growth of the industry is substantial.

The supply of this material varies little from year to year. It could therefore provide more stability than virgin oil production. Its price is highly variable, but much lower than virgin vegetable oil. The quality of the recovered oil appears to be adequate for the production of bio-diesel of reasonable quality.

The properties of beef tallow are less favourable than those of vegetable oil. However, if the control measures introduced to curtail the spread of BSE continue to provide a supply of very low-value or waste tallow, at least 50,000 tonnes of this material could be available for bio-diesel production within the EU, and 2,000-3,000 tonnes in Ireland.

## **CONCLUSIONS**

The following are the main conclusions arising from this work:

- It is possible to produce bio-diesel of acceptable quality from low-grade tallow, including that from BSE risk organisms. Some modifications and additions to the conventional esterification process are required.
- Esters of waste oils and fats oils give a performance similar to rape methyl ester in vehicles. Problems of high melting point can be overcome by mixing with mineral diesel. Tallow ester requires careful filtration.
- Waste oils and fats are available in Ireland and throughout the EU in sufficient quantities to greatly increase bio-diesel production. Their price is much lower than that of virgin oil; in

the case of BSE risk tallow, a high disposal cost would be avoided. The quality of these materials is generally acceptable for bio-diesel production.

## **PUBLICATIONS**

**Fröhlich, A. and Rice, B. 1996.** Bio-diesel from waste cooking oil. Proceedings of the 2<sup>nd</sup> activity meeting of the International Energy Agency, Vienna, November, 1995, 11-18.

**Rice, B. 1999.** Waste oils and fats as bio-diesel feedstocks: an assessment of their potential in the EU. ALTENER Programme, NTB-NETT Phase III, Task 5 Report.

**Rice, B., Bulfin, M., Kent, T., Fröhlich, A. and Lenehan, J.J. 1997.** Potential for energy production from agricultural and forest biomass in Ireland. Teagasc, Oak Park.

**Rice, B., Fröhlich, A. and Leonard, R. 1997.** Industrial possibilities of bio-diesel based on used frying oil in Ireland. In: Workshop "Valorisation of used frying oils and fats in the Benelux." Brussels, Dec. 10, 1997, 1-5.